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IS 15080 (2001): 6-Bromo-2, 4-Dinitroaniline, Technical - Spcification [PCD 9: Organic Chemicals Alcohols and Allied Products and Dye Intermediates]

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भारतीय मानक

6-ब्रोमो-2,4-डाइनाइट्रोएनीलीन, टेकनीकल — विशिष्टि

Indian Standard

6-BROMO-2,4-DINITROANILINE, TECHNICAL — SPECIFICATION

ICS 71.080.80

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BUREAU OF INDIAN STANDARDS
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 110002

December 2001

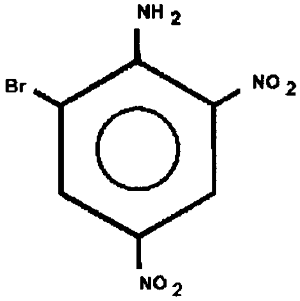
Price Group 2

Dyes Intermediate Sectional Committee, PCD 11

FOREWORD

This Indian Standard was adopted by the Bureau of Indian Standards, after the draft finalized by the Dyes Intermediate Sectional Committee had been approved by the Petroleum, Coal and Related Products Division Council.

6-Bromo-2,4-dinitroaniline ($C_6H_4O_4N_3Br$) is an important intermediate, used in manufacture of disperse dyes. It is represented by following structural formula:



6-Bromo-2,4-Dinitroaniline
(Molecular mass 262)
CAS Registry No. [1817-73-8]

The composition of the Committee responsible for formulation of this standard is given in Annex B.

For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated expressing the result of a test or analysis, shall be rounded off in accordance with IS 2 : 1960 'Rules for rounding off numerical values (*revised*)'. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

Indian Standard

6-BROMO-2,4-DINITROANILINE, TECHNICAL — SPECIFICATION

1 SCOPE

This standard prescribes the requirements and methods of sampling and test for 6-bromo-2,4-dinitroaniline.

2 REFERENCES

The following Indian Standards contain provisions which through reference in this text constitute provisions of this standard. At the time of publication, the editions indicated were valid. All standards are subject to revision and parties to agreement based on this standard are encouraged to investigate the possibility of applying the most recent editions of the standards given below:

IS No.	*Title*
1070 : 1992	Reagent grade water (*third revision*)
5299 : 2001	Methods of sampling and tests for dye intermediates (*first revision*)

3 REQUIREMENTS

3.1 Description

The material shall be orange to brownish yellow dry powder and shall be free from visible impurities.

3.2 The material shall also comply with the requirements given in Table 1.

Table 1 Requirements for 6-Bromo-2,4-Dinitroaniline

(*Clause* 3.2)

Sl No.	Characteristics	Requirements	Method of Test
(1)	(2)	(3)	(4)
i)	Assay, percent by mass, *Min*	98.0	Annex A
ii)	Melting point, °C *Min*	148	Cl 9 of IS 5299
iii)	Matter insoluble in methanol, percent by mass, *Max*	0.2	Cl 11 of IS 5299
iv)	2,4-Dinitroaniline, percent by mass (on 100% basis), *Max*	1.0	Annex A

4 PACKING AND MARKING

4.1 Packing

The material shall be packed as agreed to between the purchaser and the supplier.

4.2 Marking

4.2.1 Each container shall be securely closed and shall bear legibly and indelibly the following information:

a) Name of the material;
b) Indication of the source of manufacture;
c) Net mass;
d) Lot or batch number; and
e) Month and year of manufacture.

4.2.2 *BIS Certification Marking*

4.2.2.1 Each container may also be marked with the Standard Mark.

4.2.2.2 The use of the Standard Mark is governed by the provisions of the *Bureau of Indian Standards Act*, 1986 and the Rules and Regulations made thereunder. The details of conditions under which the licence for the use of the Standard Mark may be granted to manufacturers or producers may be obtained from the Bureau of Indian Standards.

5 SAMPLING

5.1 The method of drawing representative samples of the material shall be as prescribed in **4** of IS 5299.

5.2 Number of Tests

Tests for description, melting point, 2,4-dinitroaniline and assay shall be done on each sample. Test for matter insoluble in methanol shall be performed on the composite sample.

5.3 Criteria for Conformity

5.3.1 *For Individual Samples*

The lot shall be declared as conforming to the requirements of the description, melting point, 2,4-dinitroaniline content and assay, if each of the individual test results satisfies the relevant requirements given at **3.1** and in Table 1.

5.3.2 *For Composite Samples*

For declaring the conformity of the lot to matter insoluble in methanol tested on the composite sample, the test result shall satisfy the relevant requirement given in Table 1.

6 TEST METHODS

6.1 Tests shall be conducted according to the methods prescribed and as indicated in col 4 of Table 1.

6.2 Quality of Reagents

Unless specified otherwise, pure chemicals and distilled water (*see* IS 1070) shall be employed in tests.

NOTE — 'Pure chemicals' shall mean chemicals that do not contain impurities which affect the results of analysis.

ANNEX A

[*Table* 1, *Sl No.* (i) *and* (iv)]

GAS CHROMATOGRAPHIC ANALYSIS FOR 6-BROMO-2, 4-DINITROANILINE AND 2, 4-DINITROANILINE

A-0 GENERAL

The contents of 6-bromo-2, 4-dinitroaniline and 2 , 4-dinitroaniline are determined by gas chromatographic analysis. Reference may be made to IS 5299 for details of GC test method to be followed. However, necessary details of test conditions are given here for guidance only.

A-1 APPARATUS

A-1.1 Any suitable gas chromatograph and column capable of being operated under conditions suitable for resolving the individual constituents into distinct peaks may be used. The conditions for a typical chromatographic analysis of 6-bromo-2 , 4-dinitroaniline is given below:

Sample	6-bromo-2,4-dinitroaniline (20 percent solution in dimethyl formamide)
Injection quantity	0.8 microlitre
Column	
Material	glass
Length	2m
Orifice	0.3 cm ID
Packing material	10 percent OV 101 on Chromosorb WHP, 80-100 mesh
Carrier Gas	Nitrogen
Conditions	
Carrier flow rate	30 ml/min
Oven temperature programming to	150°C at 8°C/min 250°C, hold 20 minutes
Injection port temperature	330°C
Detector	
Type	F. I. D
Temperature	330°C

A-1.2 Interpretation of Chromatogram

The peaks for 2,4-dinitroaniline and 6-bromo-2,4-dinitroaniline are shown at 14.33 and 15.24 minutes respectively. A typical chromatogram under the conditions given above is shown in Fig. 1.

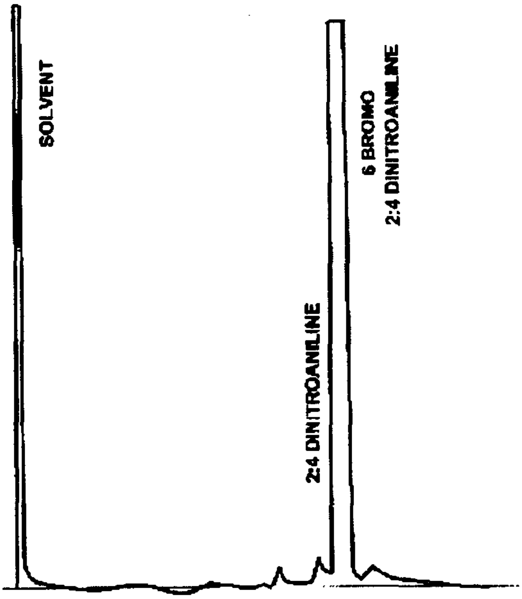


FIG. 1 A TYPICAL CHROMATOGRAM OF 6-BROMO-2,4-DINITORANILINE TECHNICAL

A-2 CALCULATION

Follow the procedure given below for calculation when internal standard method is employed.

Percent by mass of 6-bromo- 2, 4-dinitroaniline =

$$\frac{\text{Concentration of standard} \times \text{Area of peak in the sample} \times \text{Strength of standard}}{\text{Concentration of sample} \times \text{Area of peak of standard}}$$

Similarly other fractions can also be calculated.

ANNEX B

(*Foreword*)

COMMITTEE COMPOSITION

Dyes Intermediate Sectional Committee, PCD 11

Organization	*Representative(s)*
Atul Limited, Valsad, Gujarat	Dr H. Kaiwar (***Chairman***) Shri H. B. Dhuvad (*Alternate* I) Dr J. G. Desai (*Alternate* II) Shri A. R. Desai (*Alternate* III)
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